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Apparatus for Safe, Oxygen-Filled Flask Combustion

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Abstract

Two approaches for the ignition of samples in oxygen-filled flask combustions are described; namely, electrical heating of a wire and radiant energy. These approaches reduce the danger of injury from explosion by permitting the sample to be ignited while the upright flask is enclosed by a shield. For either approach, the conical flask is fitted with a socket joint. The electrically fired assembly consists of (*1*) a ball joint to which is attached a platinum spiral holder tapered at the bottom so that the sample in a capsule or paper is simply dropped into the holder, and (*2*) an easily assembled firing system requiring a variable transformer, a contact switch, and a heavy rubber electric slug which pits over the length of the ball joint. For ignition by radiant energy, beamed from a special light bulb, the sealed ball joint has a glass hook from which is suspended a platinum basket. For rapid ignition, a black paper flag is inserted into the basket with the sample. The ball and socket joint in either approach is held in place by a spring-type clamp.

Many designs of closed-flask combustion apparatus have been proposed since Hempel's publication[4] in 1892. Most of these have been concerned more with the shape of the flask than with features which stress safety in combustion. However, remote control safety devices have been described by Martin and Deveraux,[6] Juvet and Chiu,[5] Cheng and Smullin,[1] and Halsam, Hamilton, and Squirrell.[3]

Three of the systems[1,5,6] ignite the sample by passing an electric current through a high-resistance wire which either heats the sample to ignition temperature directly or ignites a paper fuse which in turn ignites the sample. Halsam et al. used a high-frequency tester to cause a spark to jump across two terminals and ignite a wick leading to the sample basket.

It is the authors' belief that safety precautions are essential. When a combustible material is ignited in an atmosphere of oxygen in a closed system, an explosion is possible. The probability that an explosion will occur under the conditions of the analysis is quite small. However, an error in charging the flask or lack of knowledge of the combustion characteristics of a sample can lead to an explosion with possible injury to the analyst. Explosions (or implosions) have been not only reported but experienced. Fortunately, a remote-control safety apparatus was being used when the explosions occurred in this laboratory.

Experience has shown the authors that the apparatus of Martin and Deveraux has some weaknesses which tend to develop after extended use. The electrical contacts in the head of the ignition unit may become loosened, the fuse wire may come loose from the posts, the glass may crack around the platinum seal, or finally, the wire in the mesh sample holder may become brittle and break. To try to overcome some of these faults, a modified, electrically-fired, remote-control apparatus was designed and constructed in this laboratory. After this apparatus had been in use for several months, it occurred to the authors that a more simply constructed flask could be used if the sample could be ignited by a light beam. A proximity lighter, sometimes used to light cigarettes and cigars in automatic smoking machines, was found to be suitable and a safety apparatus using a proximity lighter was constructed. The description of these two apparatuses follows.

Safety Apparatus Using Electric Ignition (Fig. 1)

Flask

The most popular combustion flask has been an Erlenmeyer flask with a standard taper joint and with the sample holder and ignition unit sealed in the stopper. The Erlenmeyer flask was considered the most desirable shape but a 35/25 ball and socket closure with spring-type clamp was chosen as a more convenient means of sealing the flask during combustion. The ball stopper was constructed with a 13-mm. o.d. tube extending below as well as above the joint.

Lead glass was sealed into the tube below the joint and tungsten wires were sealed in this glass so that they extended about 1 cm. below the seal. Insulated copper wires which had been attached to the

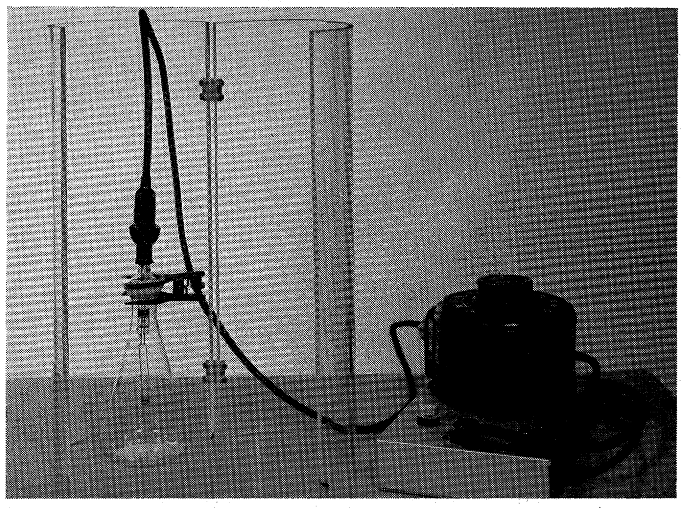

Fig. 1. Closed flask combustion apparatus using electric ignition.

tungsten wires were lead out through the top tube where they were connected to an electric plug. This rubber plug, the type commonly used on heavy service cords, had a long sleeve which fit snugly over the top tube of the stopper.

The sample holder was made of 18-gage platinum—rhodium wire wound into a cylinder, 8 mm. i.d. and 10 mm. high, with a spiral bottom, and with leads about 5 and 6 cm. long which were spot-welded to the tungsten wires sealed in the stopper.

Firing Mechanism

The sample holder also serves as the source of heat necessary to ignite the sample. Because the resistance of the 18-gage wire is so low, a relatively large amount of current is required to heat the wire sufficiently to cause ignition. The current is supplied by a 10-amp. variable transformer (Variac*) set to deliver 30 v. Although the current drawn is about 50 amp., the short time required, 1 to 2 sec., prevents any damage to the transformer. Momentary overloads of as much as 1000% will not harm the transformer used. A fuse and

* Mention of a specific product does not constitute endorsement of that product by the Department of Agriculture over similar products not named.

push-button switch on the primary side of the transformer complete the electrical equipment.

Safety Shield

A split cylinder shield was constructed from $^1/_4$-in. thick Plexiglas by cutting out two rectangular pieces 28 cm. by 45 cm. and placing these over a cylindrical form 18 cm. in diameter in an air oven. The temperature of the oven was increased slowly until the Plexiglas softened and took the shape of the form. The two halves of a cylinder thus formed were hinged top and bottom on one side, and when standing on end on the bench top constituted the shield.

Discussion

The ball and socket closure on the Erlenmeyer flask has proven very satisfactory. There is no tendency for the joint to stick as is the case with standard taper joints. The joint should be moistened but it is not necessary to invert the flask when a clamp is used.

Erlenmeyer flasks with special socket joints having a raised flange to form a shallow cup around the ball joint were constructed. Water was placed in the cup to form a seal during combustion and to rinse the stopper when the flask was opened. The cup had to be so shallow to accommodate the clamp that it did not hold enough water to rinse the joint efficiently. It was also found that the water seal was not necessary, so the special joints were abandoned.

Five hundred-milliliter flasks normally are used, but 300-ml. or liter flasks can be substituted using the same stopper assembly. The sample, in either a capsule or folded paper, may be placed in the platinum wire cup. When capsules are used they must be heated to ignition temperature in 1 to 2 sec. to prevent the expanding air in the capsule from blowing the capsule apart before the sample is ignited. This will probably be critical only when the sample has an appreciable vapor pressure below its ignition temperature or that of the capsule. Under the conditions used (30 v.) heating is so rapid that the capsule is ignited before the gases are expanded sufficiently to separate the two parts of the capsule.

A sample holder of 20 or possibly 22 gage platinum alloy wire should be sufficiently rigid to hold its shape under normal handling. This would have the advantage of decreasing the amount of current required to heat the sample rapidly to ignition temperature and permit the use of a 5-amp. variable transformer.

In the original design the tungsten leads were extended to the platinum sample holder. This was satisfactory for halogen and sulfur analysis but interfered with the phosphorus determination by the molybdenum blue reaction.

Safety Apparatus Using Proximity Lighter (Fig. 2)

Flask

A 500-ml. Erlenmeyer flask with 35/25 socket joint was selected, although a 300-ml. flask also can be used. A stopper, made from the ball joint previously described, with a glass rod sealed to the lower side, was held in place by a conventional spring clamp. The end of

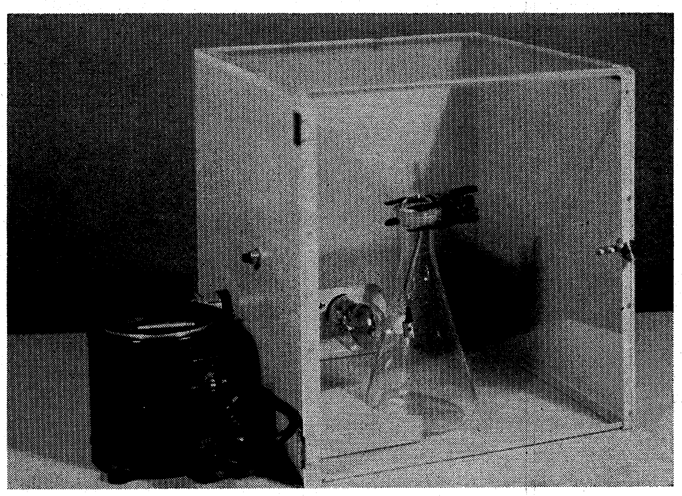

Fig. 2. Closed flask combustion apparatus using light ignition.

the glass rod was shaped into a hook and the rod was bent to place the hook near the wall of the flask when the ball joint was seated in the socket. To position the sample holder correctly, the hook on each stopper must be the same distance from the bottom of the joint (approximately 65 mm.). The sample holder was made from a piece of sheet platinum cut in the shape of a cross, then bent to form a cup 8 mm. i.d. by 13 mm. high with a handle 1 cm. wide and 3 cm. long.

Holes were cut in the bottom and walls of the cup to provide ready access of oxygen to the burning sample. A hole at the top of the handle permits hanging the cup on the glass hook. The sample in either a capsule or folded paper is placed in the cup of the holder.

Firing Mechanism

The source of the light beam used to ignite the sample is a Sylvania Tru-Flector projection lamp, DLG, T-14 Lo-volt, which is operated by a 5-amp. variable transformer set at 20 v. This lamp focuses the light on a spot about 1 cm. in diameter about 2 cm. from the lamp. The bent glass rod enables the sample holder to be placed at or very near the focal point of the lamp. To speed ignition, a black paper fuse is inserted in the sample holder so that it protrudes above the cup. The light beam is aimed at the black fuse which ignites in 1 to 2 sec. The fuses can be made from Whatman No. 29, cut in strips about 6 mm. wide, or from white filter paper blackened with india ink or Sudan Black B dye. White filter paper fuses require an excessively long time to ignite.

Safety Shield

To protect the operator in case of explosion, combustions are carried out with the proximity lighter and flask in a box about 1 ft./side. The top and hinged front door are of $^1/_4$-in.-thick Plexiglas while the sides and back are of 22 gage metal. The lamp and a push-button switch are fastened to one side of the safety box. At the bottom of the box below the lamp is a wooden block with a semicircular cutout 10 cm. in diameter which aids in positioning the flask with respect to the lamp and prevents the flask from hitting the lamp. Except for the wooden block, the bottom of the box is open.

Discussion

The use of a proximity lighter permits the flask construction to be more simple than is required for electrically fired safety units. The Erlenmeyer flask with ball and socket closure held by a suitable clamp eliminates the need to invert the flask. In addition, the detachable sample holder can be dropped into the absorbing solution after sample combustion for phosphorus analysis.[2] The precision and accuracy obtained with either of the apparatus described have been as good as those obtained with the apparatus of Schöniger[7,8] or Martin and Deveraux.[6]

References

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Discussion

F. W. Cheng (*Atlas Chemical Inc., Wilmington*): Do you know if there have been any explosions or breakage during flask combustion experiments?

C. L. Ogg: There have been a few cases reported where the sample being analyzed has exploded on ignition with sufficient violence to break the flask. We have had one explosion for which we have no other explanation.

G. Grimes (*Galbraith Labs., Knoxville*): Do you have trouble with incomplete combustion of cellulose capsules?

C. L. Ogg: None that we are aware of; however, with very volatile samples some may escape complete combustion.